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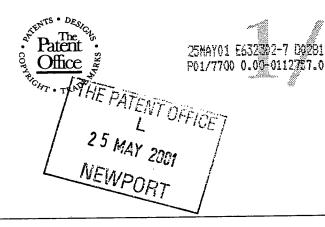
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#### Patents Form 1/77

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C505.00/U

Patent application num (The Patent Office will fill in ..... 0112757.0

25 MAY 2001

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Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

Unilever PLC PO Box 68 Unilever House Blackfriars London

EC4P 4BQ

1000162-2001

England

Title of the invention

Drink Product

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

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Patent Department.

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6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

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Date of filing (day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Patents ADP number (if you know it)

Number of earlier application

Date of filing (day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

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b) there is an inventor who is not named as an applicant, or

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## Patents Form 1/77

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Clare Matthews - 01223 355477

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#### TITLE: DRINK PRODUCT

#### Field of the Invention

This invention relates to a drink product comprising a container holding a water-based beverage under gaseous pressure in a headspace above the beverage.

## **Background to the Invention**

In known carbonated drink products, a water-based beverage is packaged with carbon dioxide under a gauge pressure greater than one atmosphere, typically several atmospheres gauge pressure. The beverage is then exposed briefly to atmospheric pressure before a closure is applied to the container holding the beverage. The beverage is therefore supersaturated with carbon dioxide gas when the beverage is brought back to atmospheric pressure, and during consumption. When the beverage is brought back to atmospheric pressure, and during consumption, some of the excess carbon dioxide is liberated in the form of bubbles or effervescence. The bubbles rapidly grow in size and produce a prickly sensation in the mouth of the consumer.

Also, the presence of a large amount of carbon dioxide in solution in the beverage produces a distinctive taste of carbonic acid. Many consumers find both the prickly sensation and the taste of carbonic acid to be unpleasant.

It is known, for example from JP 64-27458, to provide a health drink product having a beverage with oxygen dissolved therein under pressure. However, oxygen is very sparingly soluble in water and in the known health beverage nearly all of the oxygen

packaged with the beverage escapes when the beverage container is opened, with the result that there is almost no effervescence on consumption. The invention aims to provide a drink product which gives effective effervescence on consumption but avoids the disadvantages of the prickly sensation and the taste of carbonic acid which result from the use of carbon dioxide as the effervescent agent.

#### Summary of the Invention

According to the invention a drink product comprises a container holding a water-based beverage under gaseous pressure in a headspace above the beverage, and a normally-closed valve openable to allow discharge of the beverage from the container, wherein the beverage has dissolved therein, under a gaseous pressure in the headspace of at least 2.5 atmospheres gauge at  $10^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ , a sparingly soluble gas selected from oxygen, nitrogen, noble gases and mixtures thereof. Such a sparingly soluble gas, when packaged with the beverage in the container above the threshold pressure of 2.5 atmosphere gauge, produces on consumption at a serving temperature (generally between just above 0°C and room temperature, and preferably in the range 5°C to  $10^{\circ}\text{C}$ ) effervescence caused by the generation of very small bubbles of the sparingly soluble gas, which do not give the prickly sensation produced by effervescence of a more soluble gas such as carbon dioxide: instead, the sensation is described as smooth or silky. The fine effervescence produced may also enhance flavour delivery from a flavoured beverage. If the beverage is transparent, for example water or a flavoured water, the effervescence of the sparingly soluble gas gives the beverage a fine, smoke-like appearance on consumption.

The sparingly soluble gas may be wholly oxygen or wholly nitrogen, and in the preferred embodiment there is none, or substantially none, of any more soluble gas (such as carbon dioxide) in the beverage. In this case, the beverage is free from the taste of carbonic acid.

The valve may be an aerosol valve, having a movable valve member spring-biased to a closed position, the valve member being translatable or tiltable to open the valve to allow discharge of the beverage under pressure.

## **Brief Description of the Drawings**

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The invention will now be further described, by way of example, with reference to the accompanying drawings, in which:

Figure 1a is a diagrammatic side view of a first embodiment of drink product according to the invention.

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Figure 1b is a fragmentary view, to an enlarged scale, showing the structure of a valve of the drink product of Figure 1a,

Figures 2 and 3 are side views corresponding to Figure 1 but showing second and third embodiments of the invention, respectively,

Figure 4 is a graph indicating acceptable pressures for the drink products of Figures 1a, 2 and 3,

Figure 5 is a view of an assembly used to test the effervescence of the drink product of Figure 2,

Figure 6 is a graph illustrating the variation of bubble size with varying partial pressure of oxygen in the drink product of Figures 1a, 2 and 3, and

Figure 7 is a table of test results.

#### **Detailed Description of the Preferred Embodiments**

The drink product illustrated in Figure 1a has a hollow container 2 capable of withstanding an internal gauge pressure of at least 2.5 atmospheres. The container 2 holds a water-based beverage 1 having dissolved therein some of a sparingly soluble gas (oxygen, nitrogen, one or more noble gases (particularly helium, neon and kyrpton) or a mixture thereof) within a headspace 5 above the beverage 1. The container 2 has a neck to which is fitted a normally closed valve 3 which is a high-throughput aerosol valve for low viscosity products. A dip tube 4 extends downwardly from the valve 3, through the headspace 5 and into the beverage 1 to allow the latter to be expelled when the valve is opened with the container 2 upright.

The valve 3, shown in greater detail in Figure 1b, has a valve housing 15 the lower end of which is attached to the upper end of the dip tube 4 shown in Figure 1a. The upper end of the housing 15 has a gasket 19 forming a valve seating which co-operates with a movable valve member constituted by a stem 17 biased by a spring 20 to a closed position in which the stem 17 engages the gasket 19. A further gasket 16 and an annular ring 18 seal the valve 3 to the neck of the container 2.

To dispense the beverage, the user tilts or depresses the stem 17 (or another element attached to the stem 17) so as to overcome the bias of the spring 20 and allow the beverage to pass between the stem 17 and the gasket 19. The beverage may be dispensed into another container for drinking but is preferably dispensed directly into the consumer's mouth.

The drink product shown in Figure 2 is similar to that of Figure 1 but is devoid of a dip tube 4, so the drink product of Figure 2 is usable with the container 2 in an inverted position.

In the embodiments of Figures 1a and 2, the beverage 1 is placed in the container 2, which may then be flushed with the sparingly soluble gas mixture. The container 2 is then sealed with the valve 3, and more of the gas mixture is added through the valve until the pressure in the headspace 5 has reached its required value, which is more than 2.5 atmospheres gauge. When the valve is then opened for operation the pressure in the headspace 5 expels the beverage through the valve 3. Because of this, a substantial amount of headspace is initially present (unless the pressure is very high). Preferably, the headspace 5 occupies between 50% and 80% of the capacity of the container 2.

In the drink product of Figure 3, the dip tube 4 is replaced by a flexible bag 6 having an upper neck sealed to the housing 15 of the valve 3. The bag 6 holds the beverage 1 under pressure from the sparingly gas in the headspace 5 which can be considerably smaller than the headspace 5 of the drink products of Figures 1a and 2. There is a second gas space 7 between the bag 6 and the container 2, and this second space 7 is filled with pressurised gas which may be the same gas (or gaseous mixture) as in the headspace 5 or may be different therefrom. This construction allows the beverage to be dispensed in any orientation of the container 2.

For the embodiment of Figure 3, the sparingly soluble gas is placed in the headspace 5, which may be very small, preferably less than 20% of the capacity of the bag 6. The space 7 between the bag 6 and the container 2 is pressurised after the valve 3 is sealed to the container, and the expansion of gas in this space provides the driving force to expel the beverage through the valve during discharge of the beverage.

In each embodiment, the pressure in the headspace 5 exceeds 2.5 atmospheres gauge, which causes the sparingly soluble gas (or mixture of sparingly soluble gases) to effervesce on discharge into the consumer's mouth or into water under test conditions. To demonstrate the effect the following experiments were carried out.

## Experiment 1

It was found that the fine smoke-like effervescence is produced from a beverage that has an initial pressure in the headspace of 2.5 atmospheres or more, for all mixtures of oxygen and nitrogen.

This was tested by preparing products with varying pressures and varying proportions of oxygen in an oxygen-nitrogen mixture. These products were then discharged under water in front of a piece of black plastic, and the smoke-like effervescence was observed visually.

It was found that for any given proportion of oxygen in the mixture, then at low filling pressures the effervescence was not observed, but above a critical pressure the effervescence was observed. Figure 4 shows a plot of the experimental results. The points plotted with error bars show the position of the critical filling pressure for each of the mixtures tested. Based on the results, a filling pressure of 2.5 bar or greater (that is to say, the entire region above the dashed line labelled 32 in Figure 4), produced effective effervescence.

# Experimental Protocol for the First Experiment:

For each of the proportions of oxygen in the mixture that were tested, a range of samples was prepared and tested at increasing filling pressures. Two samples were tested for each pressure to check reproducibility.

For each sample, the following protocol was observed:

(1) A strong polyethylene terephthalate (referred to as "PET") bottle of 28g mass and 520ml brimful capacity was taken, and in it was placed 300ml of tap water at  $10^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  (all temperatures in the following being measured using a

thermocouple probe). The bottle was then left to equilibrate in a temperature-controlled bath also at  $10^{\circ}C \pm 0.5^{\circ}C$ .

- (2) The proportion of oxygen gas in the oxygen-nitrogen mixture had been decided on in advance, and whichever gas was to be at a higher partial pressure in the mixture is called "gas 1". The other gas will be referred to as "gas 2".
- (3) The headspace above the water in the bottle was flushed with gas 1 for 5 to 10 seconds, and then the aerosol valve was placed quickly over the neck of the bottle, thus sealing the bottle from any loss of gas 1 or influx of air. The aerosol valve was then held firmly in place by means of a screw cap with a small hole cut in the centre in order to allow the stem of the valve to be accessible.
- (4) More of gas 1 was then added from the regulated supply of gas 1 through the stem of the aerosol valve until a pressure "p1" was reached. The bottle was then shaken to ensure that the gas inside was at the same temperature as the liquid, and the bottle was topped up to the pressure p1 with more of gas 1. This was necessary because the temperature of the gas when first injected into the bottle is somewhat higher than 10°C.
- (5) Gas 2 was then added through the stem of the valve from a regulated supply of gas 2 until a pressure "p2" was reached. Again the bottle was shaken, and then topped up with more of gas 2 to reach the pressure p2 again. Both p1 and p2 are measured in atmospheres gauge from the pressure gauge on the regulated supplies of the two gases.
- (6) The sealed bottle was then placed in a temperature controlled bath at 10°C±0.5°C for 5 minutes to equilibrate, the sample being shaken every 30 seconds to ensure good contact between the gas mixture and the water in the bottle.

(7) The bottle was then discharged under water which was at a temperature of 21°C±1°C. The stream of discharged fluid was observed as it left the bottle against a black plastic sheet, and it was noted whether or not there was a visible, fine, smoke-like effervescence.

# Results of the First Experiment

Figure 4 shows a plot of the molar fraction of oxygen present in the mixture (horizontal axis) against the gauge pressure p2 (vertical axis).

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The experimental results are for the embodiment shown in Figure 2. Each error bar in Figure 4 runs from a lower value of p2, which is the highest pressure at which the effervescence is definitely not seen, to a higher value of p2, which is the lowest pressure p2 at which effervescence is definitely seen. The true critical pressure thus lies somewhere on the error bar. If there is a pressure at which there is some doubt as to whether effervescence can be seen or not, this is marked as an "x" on the error bar. These "x" marks thus represent the best estimate of the position of the critical filling pressure.

The graph has two results drawn with a lighter weight of line and marked 30 and 31 in Figure 4. These results are shown slightly displaced in the horizontal direction, for the sake of clarity. Thus the true molar fraction of oxygen in the mixture for result 30 is 0.0, and for result 31 it is 1.0. The results labelled 30 and 31 are for a tilt operated aerosol type valve, while all the other results are for a push-down operated aerosol type valve.

## Experiment 2:

It was found that the fine smoke-like effervescence produced by mixtures of nearly insoluble gases, and which gives a smooth (as opposed to prickly) sensation to the

consumed beverage, is preserved in the presence of admixtures of a soluble gas, provided that the partial pressure (absolute) of the soluble gas above the beverage in the container does not exceed atmospheric pressure. That is to say, provided the soluble gas is not overly supersaturated at the serving pressure of one atmosphere absolute pressure.

# Experimental Protocol for the Second Experiment

The experimental set-up is shown in Figure 5. The embodiment of Figure 2 was used.

- (1) A strong PET bottle of 28g mass and 600ml brimful capacity was taken (labelled 2 in Figure 5), and in it was placed 300ml of tap water (labelled 40 in Figure 5) at 18°C±0.5°C (all temperatures in the following being measured using a thermocouple probe). The bottle was then left to equilibrate in a temperature-controlled bath also at 18°C±0.5°C.
- (2) Twelve experiments were performed, referred to in the following by letters "A" to "L" inclusive. These experiments differed in the way gas was added to this bottle, and is described immediately:

Experiment A: The valve 3 of Figure 5 was secured to the bottle 2, using in this case a screw cap 44 with a hole in the centre to admit the stem of the valve 3 (labelled 17 in Figure 1b). This procedure formed a gas-tight seal, isolating the contents of the bottle from its surroundings. In what follows, this process will be referred to as "the bottle 2 was sealed with the valve 3 and cap 44". Next, oxygen was added through the valve stem until the pressure inside the headspace 5 was 4 atmospheres gauge. The bottle was shaken, and then topped up with oxygen in the same way until the pressure inside was once more 4 atmospheres gauge.

Experiment B: The headspace of the bottle was flushed with carbon dioxide from a regulated supply of the gas, for 5 seconds, to clear the headspace of air. The bottle

2 was then sealed with the valve 3 and cap 44, and CO<sub>2</sub> was added at 4 atmospheres from the regulated supply through the stem of the valve. While the gas was added, the bottle was constantly shaken for 20 seconds, so that the carbon dioxide was close to equilibrium with the water 40.

Experiment C: As for experiment A, but before the bottle 2 was sealed with the valve 3 and cap 44, the headspace was flushed with oxygen for 5 seconds to remove the air from the headspace 5.

Experiments D and J: The bottle was flushed with oxygen and sealed as in experiment C. Oxygen gas was then added through the stem of the valve, up to a pressure of 3 atmospheres gauge in experiment D and 3.5 atmospheres gauge in experiment J. The bottle was then shaken and topped up to 3 atmospheres gauge in experiment D and 3.5 atmospheres gauge in experiment J with oxygen. Carbon dioxide was then added through the valve stem with continuous shaking for 20 seconds up to a final pressure of 4 atmospheres gauge in both cases.

Experiment E: The headspace was flushed for 20 seconds with  $CO_2$ , while the bottle was shaken, so that the  $CO_2$  was close to equilibrium with the water 40. The bottle 2 was then sealed with the valve 3 and a cap 44, and oxygen added through the stem of the valve to a pressure of 4 atmospheres gauge. The bottle was shaken, and topped up to 4 atmospheres gauge with oxygen.

Experiments F, H and I: The bottle was flushed for 5 seconds with  $CO_2$ , then sealed with the valve 3 and cap 44. Carbon dioxide was then added through the stem of the valve, with shaking, for 20 seconds. The final pressure of  $CO_2$  was chosen to be 1, 2 and 3 atmospheres gauge for the experiments F, G and H respectively. The bottles were then topped up with oxygen to 4 atmospheres gauge in all cases, shaken and topped up to 4 atmospheres gauge again with oxygen.

Experiments G and K (which are identical): The bottle was flushed with CO<sub>2</sub> for 5 seconds to expel any air present, but was not shaken. The bottle 2 was then sealed with the valve 3 and the cap 44. Oxygen was then added through the stem of the valve up to a pressure of 4 atmospheres gauge. The bottle was then shaken, and topped up with oxygen to 4 atmospheres gauge.

Experiment L: The procedure of experiment C was followed, but using nitrogen in place of oxygen at all points.

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- (3) At this stage of the procedure, the bottles are all filled to 4 atmospheres gauge pressure, but with different mixtures of gases.
- (4) In each experiment, the bottles were allowed to equilibrate in a temperature-controlled bath at 18°C±0.5°C, for 5 minutes, with shaking every 30 seconds (as in Experiment 1).
- (5) The bottles were then inverted, and a small fraction of their contents were discharged through a flexible rubber tube (labelled 41 in Figure 5), which made a good seal with the stem of the valve 3. The rubber tube 41 was connected to a glass pipe 42, which was fixed horizontally. Most of the length of the glass pipe consisted of a flat-sided pipe of height 9mm (vertically), width 5mm and length 15cm. The other end of the glass pipe was connected to another length of rubber tube 45, which emptied into a beaker 46. The function of the flat-sided pipe was to enable the liquid discharged from the bottle to be clearly seen and imaged.

Whilst the liquid was being discharged, a video camera was used to record the appearance of the discharged fluid a distance 15cm from valve 3. This region is labelled 43 in Figure 5. A grid with lines separated by 1mm was placed behind the glass pipe to allow distances to be measured accurately from the video images.

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(6) After a short discharge through the valve, effervescence was observed in the glass pipe using the video camera. This consisted of bubbles which rose in the glass pipe. When they reached the top of the glass pipe, the bubbles coalesced into larger bubbles. However, while they were rising in the now stationary fluid, their rise velocities could be measured by counting the number of video frames required for the bubbles to rise 1mm. These velocities were converted into bubble sizes using Stokes' law for the rising of a sphere in a liquid of known density and viscosity (in our case, water at 18°C).

## Results of the Second Experiment:

From the video images, two types of rise velocities were noted: firstly the rise velocities of individually imaged bubbles, and secondly the rise velocity of the surface of the cloud of effervescence. These are referred to as a "Single" and "Multiple" rise times in the table of results (Figure 7).

The results for the mixtures of oxygen and carbon dioxide (that is to say, for experiments B, C, D, E, F, G, H, J, and K), are plotted in Figure 6. In this figure, the results from "Single" bubble rise times are plotted as filled circles, and the results from "Multiple" bubble rise times are plotted as open circles.

#### It can be observed:

- (1) That the bubble size of the effervescence increases from the valve for pure oxygen, if a large fraction of carbon dioxide is present in the gas mixture.
- (2) Experiments B to K inclusive show that if the partial pressure of the soluble gas (carbon dioxide) does not exceed one atmosphere absolute (that is to say, if the partial pressure of the sparingly soluble gas (oxygen in this case) is greater than or equal to the total absolute pressure above the beverage, minus one atmosphere),

then the effervescence size does not differ significantly from the value for pure oxygen.

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(3) Experiments A and B show that fine effervescence (like that produced by pure oxygen) is also produced for another sparingly soluble gas (nitrogen), and also a mixture of nitrogen and oxygen.

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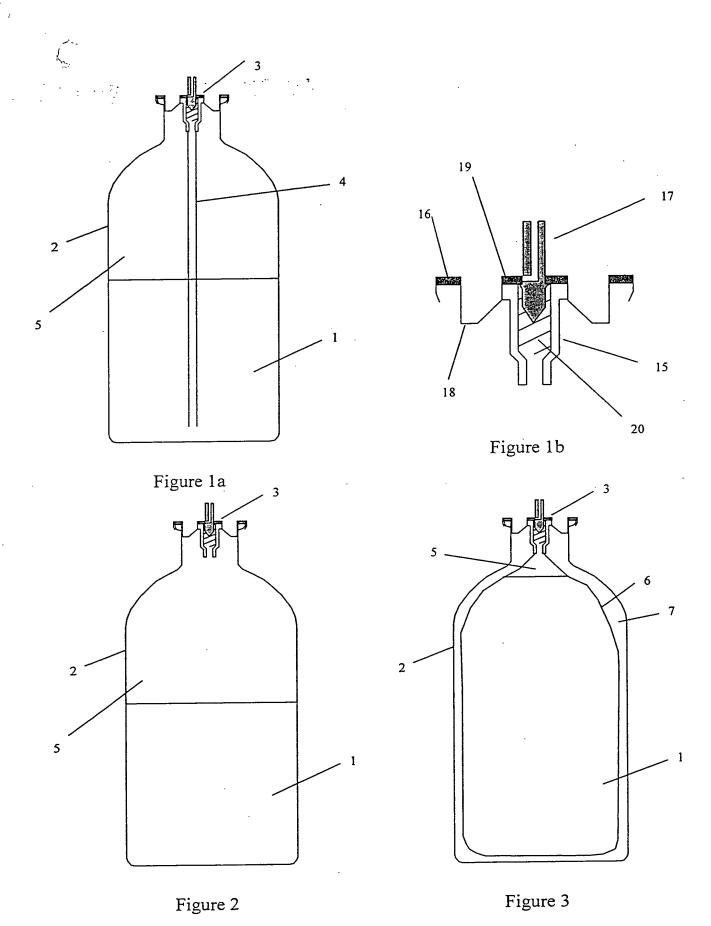
## **CLAIMS**

- 1. A drink product comprising a container holding a water-based beverage under gaseous pressure in a headspace above the beverage, and a normally-closed valve openable to allow discharge of the beverage from the container, wherein the beverage has dissolved therein, under a gaseous pressure in the headspace of at least 2.5 atmospheres gauge at  $10^{\circ}$ C  $\pm$  0.5°C, a sparingly soluble gas selected from oxygen, nitrogen, noble gases and mixtures thereof.
- 2. A drink product according to claim 1, wherein the beverage has dissolved therein a further gas which is more soluble in water than oxygen or nitrogen and which has a partial pressure in the headspace not exceeding one atmosphere absolute at 18°C.
- 3. A drink product according to claim 2, wherein the further gas is carbon dioxide.
- 4. A drink product according to claim 1, wherein there is none, or substantially none, of any gas in the beverage more soluble in water than oxygen or nitrogen.
- 5. A drink product according to any of the preceding claims, wherein the sparingly soluble gas is wholly oxygen.
- 6. A drink product according to any of claims 1 to 5, wherein the sparingly soluble gas is wholly nitrogen.
- 7. A drink product according to any of the preceding claims, wherein the valve has a movable valve member spring-biased to a closed position, the valve member being translatable or tiltable to open the valve to allow discharge of the beverage under pressure.

**Abstract** 

# TITLE: DRINK PRODUCT

A drink product has a container (2) holding a water-based beverage (1) with oxygen and/or nitrogen dissolved therein under a pressure of at least 2.5 atmospheres gauge. The container has a valve (3) openable to allow discharge of the beverage which effervesces on release from the container.



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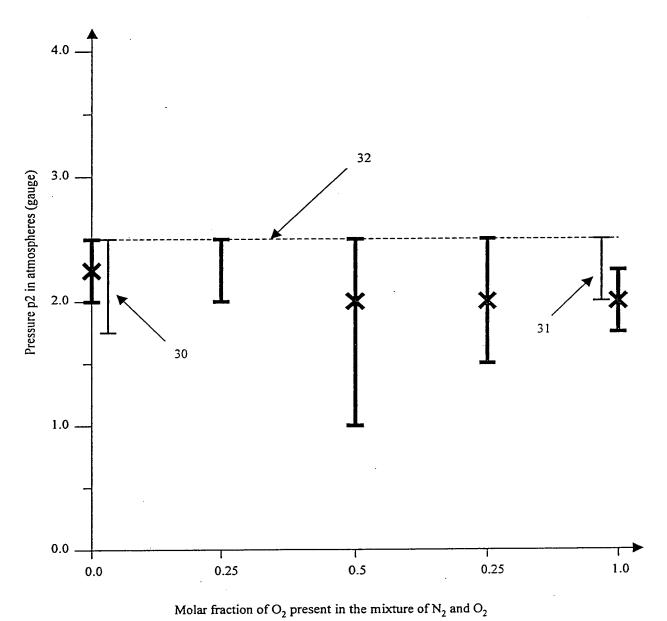


Figure 4



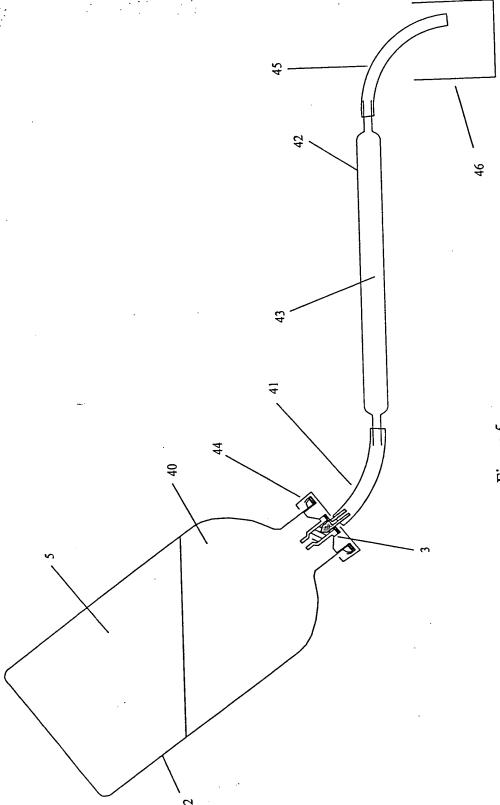


Figure 5

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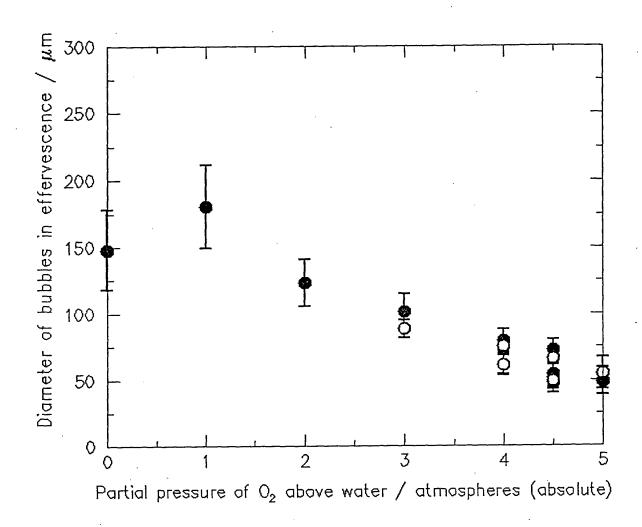


Figure 6



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Experiment	Inferred partial pressure of Oxygen above the liquid '40' in atmospheres (absolute)	Type of rise velocity measured	Number of readings taken	Bubble diameter in effervescence, deduced from Stokes' law, in micrometres
A	4.2	Single	9	48±17
A	4.2	Multiple	6	39±3
В	0.0	Single	9	148±30
C	5.0	Single	14	49±10
С	5.0	Multiple	3	55±12
D	4.0	Single	33	80±8
D	4.0	Multiple	8	75±5
E	4.0	Single	25	61±8
E	4.0	Multiple	11	61±7
F	3.0	Single	37	101±13
F	3.0	Multiple	7	88±7
G	4.5	Single	29	51±11
G	4.5	Multiple	9	48±5
Н	20.	Single	28	123±18
I	1.0	Single	12	181±31
J	4.5	Single	52	72±8
J	4.5	Multiple	11	66±5
К	4.5	Single	45	54±11
K	4.5	Multiple	10	49±5
L	0.0	Single	11	31±2
L	0.0	Multiple	12	28±3

Figure 7

Table 1: Results of experiment 2.

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